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#### SANITIZED VERSION OF HISTORY AND PROGRESS OF URANIUM CONTROL FOR K-25 (Sanitized Version of CRD Document # A-3682, dated 2/13/47)

Compiled by
S. G. Thornton
Environmental Management Division
OAK RIDGE K-25 SITE
for the Health Studies Agreement

July 1995

Oak Ridge K-25 Site
Oak Ridge, Tennessee 37831-7314
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
for the U.S. DEPARTMENT OF ENERGY
under Contract DE-AC05-84OR21400

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Mr. C.N. Rucker, Jr.

Date: February 12, 1947

### HISTORY AND PROGRESS OF URANIUM CONTROL FOR K-25

The Uranium Control and Inspection Department was organized by the writer at your suggestion to centralize the responsibility for expediting reliable over-all uranium accountability for this plant, augmenting security measures to prevent diversion of uranium and safeguarding this plant from radiation hazards.

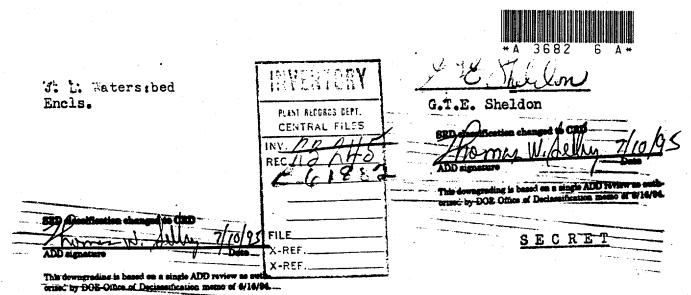
The enclosed report, "History and Progress of Uranium Control for K-25" compiled by R.W. Levin and J.L. Waters, presents the progress made by the Uranium Control and Inspection Department from the Department's formation in December 1946 to February 1, 1947, on the following major problems:

- 1. Uranium material handling and accountability;
- 2. Consumption of TF6 on plant surfaces;
- 3. Diversion control and special hazards.

In order to present a coherent picture for those persons who are not thoroughly familiar with the background of the problems a summary of the organization and technical history is included. To aid in formulating, revising or shifting the emphasis on the bases and policies that we are using, the plans for the immediate future are described.

During the period from December to February, major improvements have been made in the plant to reduce the possibility of diversion, and recommendations made by the District Engineer concerning special hazards have been placed into effect. Obtaining a reliable uranium accountability system and demonstrating the reliability is a long range problem. However, it is believed that proper emphasis is now being placed on this problem to achieve the desired results.

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# URANIUM CONTROL AND INSPECTION DEPARTMENT

Report Number A-3682

Compiled By: R. W. Levin . J. L. Waters

## HISTORY AND PROGRESS OF URANIUM CONTROL FOR K-25

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# CARBIDE AND CARBON CHEMICALS CORPORATION URANIUM CONTROL AND INSPECTION DEPARTMENT

### HISTORY AND PROGRESS OF URANIUM CONTROL FOR K-25

Date February 1, 1947
Compiled by R. W. Levin
J. L. Waters

#### INTRODUCTION

The maintenance of adequate security measures is probably the most difficult problem confronting the operator of the Uranium Gaseous Diffusion Plant. Security has several meanings which require many different measures for proper control. One type of security is concerned with information affecting the national defense. A second type of security is concerned with preventing diversion of fissionable material as specified in the Atomic Energy Act of 1946 and according to the principles of the American proposals for International Control of Atomic Energy. Complete control of "bomb material" is one of the most important security measures that can be taken before international control is achieved and it is the most important measure when international control is achieved.

Technological national control and inspection of a plant of this type includes such measures as guarding and restricting areas and providing locks and alarms to prevent diversion, in somewhat the same manner as other valuable materials are secured, such as diamonds or money. Aside from the value of "bomb material" due to its scarcity, its actual cost is also very high. No system yet proposed can completely insure against all diversion. In this plant the high concentration of X, the small weight of material which is of military significance, and the large number of sources of high concentration X in the plant area make inspection and policing of uncertain dependability. The principal burden of detecting if diversion has occurred and determining the amount, falls on material accounting. Reliable accounting for a loss of less than 20 kg. of X per year in this plant alone is extremely difficult. The material accounting system for this plant has been designed to detect losses or diversion of 20 kg. of X per year at concentrations greater than 2360001% and 35 kg. of X of any concentration. Unaccounted for losses greater than these amounts are considered to be dangerous to national security. It is essential that allowable losses of various concentrations of X for a given period of time be determined from an over-all standpoint in order to plan an effective control and inspection system.

The greatest apparent loss of X in the Gaseous Diffusion Plant is due to consumption (deposition of solid uranium compounds on plant surfaces). Consumption cannot be directly measured without removing equipment and destructively analyzing the barrier and acid washing the other surfaces. However, attempts are being made to estimate the rate of consumption from laboratory data, material balances, examination of plant equipment and tests on isolated

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units of the plant. It is estimated that the present rate may be as high as 25% of the production rate of X.

The reasons for an accurate uranium accounting system, in addition to detecting diversion, are:

- 1. To obtain measurements of the production of the plant.
- 2. To obtain over-all consumption estimates.
- 3. To detect accumulations of uranium in the plant which may exceed the critical mass.

There are many technological control and inspection problems which are not adequately solved to date. These problems are unique in that accuracies and precisions are required for measurements of weights, pressures, concentrations of contaminants, concentration of X in T, etc., that are not required or obtained in any other chemical process. The U. N. Atomic Energy Commission's Report to the Security Council accurately sums up the value of the present accountability system. "At present, it is not possible to place reliance on the method of obtaining a material balance of uranium isotopes in the case of isotope separation plants. This is one of the important reasons why there must be international control of such plants by a director or manager and why the management must be established by and be responsible to the international control agency. Even if the meterial balance could be greatly improved, the inherent danger of the operation would still require management by the international control agency.

However, there are many reasons for optimistically assuming that a sufficiently reliable material balance of X can be obtained and demonstrated in from six months to a year for this plant under current operating conditions.

### ORGANIZATION

The Uranium Control and Inspection Department was formed early in December 1946 to centralize the responsibility for handling and coordinating these many related problems which are necessary for adequate control of uranium in this plant. Following are the sections set up to carry out the program, their principal responsibilities and the personnel now assigned:

### A. Coded Chemicals Section

Orders, receives, stores, transfers and keeps records for accounting of all uranium bearing materials. Supervisors, accountants, auditors, clerks and typists total 15. There are also 15 operators (hourly).

### B. Accountability Section

Prepares procedures concerning the storage, handling and accounting of uranium bearing materials. Makes engineering studies for improving the precision and accuracy of material balances. Ten engineers and three computers are assigned.

### C. Consumption Section

Makes engineering studies and designs and coordinates the consumption programs being carried out by various plant departments. There are two engineers assigned; however most of this work is done by other service groups.

### D. Diversion Control Section

Makes engineering studies and prepares recommendations concerning the diversion control policies and practices. There are three engineers assigned. The Plant Protection Division is of course staffed to carry out the responsibilities for over-all security.

### E. Special Hazards Section

Makes studies, investigations and recommendations on all problems concerning radiation and critical mass. A physicist and an engineer are assigned. The Director of Research, Dr. Beck, is consulted frequently on special hazards problems.

The Uranium Control and Inspection Department functions as a staff organization responsible to the Assistant Plant Superintendent.

Meetings are held weekly with all persons in the plant principally responsible for the many problems concerned with uranium control in order to review the progress, determine priority on these problems as compared with non-uranium control problems and determine general policy. These meetings have been very helpful in creating interest and emphasizing the program.

### HISTORY OF DIVERSION CONTROL

Diversion of uranium that could be used in the fabrication of atomic weapons creates a definite hazard to national security. In the Baruch report of scientific information to the United Nations Atomic Energy Commission the maximum tolerable diversion has been defined as the loss of material required to make one atomic bomb per year or a total of five bombs over any period of time.

Recognizing their responsibility as the operator of the Gaseous Diffusion Plant, Carbide and Carbon Chemicals Corporation on February 20, 1946, approved the formation of a committee composed of Process and Engineering Division personnel with H. N. Woebcke as chairman. This committee was to formulate plans for safeguards to the K-25-27 Plants, beyond those in effect and enforced by the Plant Protection Division and the Army Security. This committee met frequently during the following three weeks and issued a report on March 15, 1946 giving a preliminary survey of the problems of diversion control at 15, 1946 giving a preliminary survey of the problems of diversion control at this plant with recommendations for changes in design, operating procedures, this plant with recommendations for changes in design, operating procedures, and security measures. The committee also recommended that a group of engineers be assigned full time to work on the problems in cooperation with Plant Protection and other Divisions.

A group of engineers under Mr. W. Beard was set up on March 22, and the original committee continued to function in an advisory capacity for some time. Several design changes were made to make it more difficult to remove material from the main process and better techniques of weighing were instituted. A summary report was published on October 15, 1946. This report, tuted. A summary report was published on October 15, 1946. This report, "Control of Process Material Diversion" by F. Mills and N. H. VanNie, outlined detailed design changes; presented plans for restriction of Plant II, where "unsafe" material is processed, including possible gate control plans; proposed an educational program; and outlined an inspection system.

In a meeting on November 4, 1946, of the Process Materials Department Council a proposed program for Diversion Control was prepared on the basis of the Mills-VanWie report. This proposed program was approved on November 6, 1946. Upon the formation of the Uranium Control and Inspection Department the diversion control program was placed under this department.

### PROGRAM AND PROGRESS OF DIVERSION CONTROL SECTION - H. H. STONER

#### Guneral Policies

Physical controls against diversion must be applied to all areas that contain fissionable material. The degree of control for each area within this plant depends on the amount and the X concentration of uranium handled. For uranium of feed concentration or less, practically no controls are needed except for conventional fences and guards at outer process gates to prevent removal of cylinders or large amounts of uranium, because small amounts are not hazardous unless these small amounts are consistently removed over very long periods of time. Many sections in this plant such as the laboratories, experimental users, and small chemical processing units, handle small amounts of uranium over the full concentration range and it is possible to have almost continuous accounting of the material. Conventional guarding systems coupled with detailed accounting are therefore surficient controls except in the few cases where special batches of material are being handled. However, since so many people are concerned in handling these small batches of uranium, rigid inspection of handling, accounting, storage and processing procedures by independent competent inspectors is needed.

Mest of the enriched uranium in this plant is in the form of a gas at a low pressure in the cascade. Uranium concentrations higher than 2360001 X are located in the upper third of the cascade (this portion is called Plant II). The principal burdens of rigid physical control therefore need to be placed on Plant II.

In areas where physical controls must be applied, the policy considered most workable is the elimination of any sources from which the uranium compounds could be diverted. A source is defined as the appendage to a process system, other than the end product removal point, through which material could be diverted. However, due to the very numerous sources in all portions of the casede (a torch or a tube cutting device is needed to remove material from almost all these sources), a policy of reducing the number of personnel having access to the sources is resorted to aid in maintaining adequate control. In addition, intensive inspection of personnel and equipment at all gates is required.

Physical controls and policing tend to be oppressive to individuals who are subjected to these measures. Furthermore, the operating personnel are the ones most likely to see a diversion attempt. Therefore, in applying security measures to a controlled area, those individuals working inside the area are a part of the responsibility of safeguarding the uranium compounds. Education of these men, who are effectively inspectors, emphasizes the need for control and alerts them to the dangers to national security if they should be negligent in their duty as the first line of defense against diversion. Total dependence upon these internal inspectors is not desirable since a diverter would most likely be a member of the operating personnel. A system of counter-inspection, performed by an independent organization, is advisable as a safeguard against possible corruption. This independent group is to function as additional inspectors to insure that no uranium material can be removed from the restricte



area; either on the person of an individual, concealed in equipment, or by other illegal methods.

#### Progress

The major effort on diversion control has been centered in Plant II. Two degrees of control have been instituted in Plant II. The most stringent control, based upon the need to reduce the number of personnel with access to sources, was the isolation of the cell floor and pipe gallery into what is known as the "RR" or doubly restrictly area. The second degree of control was the construction of a fence completely surrounding Plant II, forming the "R" or singly restricted area.

A "flying squadron" was organized on each shift to perform all operating functions within the "RA" area and to act as the internal inspectors. Boundary inspectors were provided for this doubly restricted area by the Plant Protection Division to insure that any visitor has a valid cherrance for entry and that the visitor is escorted by one of the internal inspectors. Upon departure of the visitor, he is searched to prevent concealment of uranium compounds on his person. Controls instituted to restrict the cell floor and pipe gallery include: locking surrounding doors and windows, sealing valve plates and other means of access from the operating floor, alteration of method of entry to canteen, establishment of portals of entry to the area, and construction of a loading platform across which metrials and equipment will pass for inspection. Certain operating procedures were revised to increase operational efficiency under the new system without sacrifice of security. These controls make the "RR" area man-tight according to the policy established for the present degree of security.

An outer fence has been built completely surrounding Plant II to restrict over-all access to this hazardous area. A badge exchange system has been initiated, and a system of verification for visitors will be initiated when the traffic can be reduced to a minimum by revision of procedures of the various service departments. Entry is made to the Plant II area through any of several portals after a badge exchange and check of badges by a guard.

An educational program was undertaken by both Process Operations and the Training Department. These lectures reached all Plant II personnel and were designed to demonstrate the need for diversion control, the responsibility of the operators as inspectors, the physical changes that were to be made, and the methods of operation under the new system. The program was well received by all.

A study has been initiated to determine the degree of control and inspection necessary in the 1300 area where several small chemical processes are carried out to recover uranium from contaminated materials and to convert the purified materials to TF6. Proposals for security measures in this area are almost complete. Ever-all security measures have been tightened by the Plant Protection Division, by eliminating private automobiles from plant areas, instituting more rigid package inspection at cutside gates, etc., which make diversion more difficult.

#### HISTORY OF ACCOUNTABILITY

During the early operation of the plant emphasis was placed on getting the plant into production, and refinements in operating procedures necessary for satisfactory material accounting were not in effect. Partial control of the handling and warehousing of uranium material was maintained by the Coded Chemicals Department; however, receiving, handling of contaminated materials, and accounting were responsibilities of several other groups. At this time other materials than uranium were classified as coded chemicals for security reasons, and were part of the responsibilities of the groups handling and accounting for uranium. Problems involving these coded chemicals often prevented sufficient emphasis being placed on uranium accountability.

On April 3, 1946, a report, "Accounting for T and X in the Diffusion Plant" was issued by Messrs. Benedict, Daniel and Squires. The report recommended improvements in the existing accounting and handling procedures, and was the first step in setting up a sound basis for determining uranium balances in the diffusion plant with a known reliability. The application of statistical methods to the interpretation of material accounting data as described in this report made it possible to set precision requirements on all measurements of uranium. The Process Materials Department was formed in June 1946 to carry out the program recommended in this report.

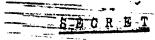
At this time the Research Laboratory started work on the determination of the absolute value of X in normal feed and the ratio of waste concentration to normal feed concentration, to a very high precision.

Using the report "Suggested Procedure for Determining Cascade Inventory", by L. Davidson, etal., April 30, 1946, as a basis, cascade inventory procedures were prepared and a comprehensive cascade inventory made on July 1, 1946. Material balances on other units handling uranium were planned, and the first laboratory balance was made during the month of August 1946.

In order to obtain adequate methods of handling and accounting, a consolidation of Material Accounting with Coded Chemicals was made on December 1, 1946.

Following the publication of the report on T and X accounting in April 1946, the Statistical Analysis Section, under Mr. C. Danial of the Process Design and Development Department, continued work on the application of statistical methods to material balances. Refinements were made in the analysis of variance and propogation of error as applied to the cascade, which resulted in a better understanding of the causes of variability of cascade measurement.

During recent months, intensive work on the analysis of T in TF6 has been carried on by the Works Laboratory. Notable success has been obtained in reaching high precision and accuracy of T analysis and the target values for precision have been bettered.



# PROGRAM AND PROGRESS OF ACCOUNTABILITY SECTION - A. DE LA GARZA

In order to determine that the yearly unaccounted for "X" in the K-25, K-27 plant is less than detailed material balances are required for all univs handling uranium.

The present plan for material balances is shown by Figure I, which gives material flows at the plant and the proposed material balance areas.

The most important of the material balances is that of the Diffusion Cascade because it is the area's largest active accumulation of enriched material, and for the present it is not possible for accounting purposes to segregate material in the cascade as to enrichment.

The required precision of the cascade balance is difficult to meet. Difficulties are encountered from the following factors:

### 1. Consumption

Unaccounted for material, as given by the caseade material balance, cannot be evaluated without a precise knowledge of the inherent cascade surface consumption of material. This consumption must be determined from independent sources or from procise T-material balances, whose high precision and consistent loss rate at steady operating conditions indicates consumption rather than diversion.

### 2. Cascade Inventory Measurements

The gaseous diffusion cascade is a complex dynamic system of 700,000 cubic feet, composed of sections of varying sizes, operating at widely varying pressure levels, temperatures, purities, X-assays, and stream flows. These operational characteristics must be interpreted to give accurate inventory changes. The necessary quantities to calculate a cascade inventory are:

- a. Size factor the calibration factor of the equipment size at a set of reference operating conditions.
- b. Relative inventory factor a measurement of carrier flow at the operating barrier permeability, barrier high side pressure, pump speed and stream light contaminant.
- c. Barrier high side pressure.
- d. Stream heavy contaminant. (C-816, etc.)
- e. Stream temperature.
- $f_*$  "X" assay.

Approximately 3000 pressure, 3000 temperature, and 3000 control valve position readings are needed for each cascade inventory.

Besides the Diffusion Cascade, the Purge Buildings, carbon and elumina traps, and approximately 65,000 cubic feet of surge and ourge systems must be inventoried.

Considerable data have been obtained to determine the precision of all measurements, and steps have been taken to reduce the uncertainty of the major sources of variability.

### 3. Operational Changes

Large operational changes of cascade inventory greatly increase the uncertainty of the balance during the period in which the changes occurred. This is brought about by biases in the equipment calibration factors and by biases in the measured quantities.

Work has been done to determine the magnitude of these biases, and in particular, work is proceeding to determine how these biases are introduced or changed by process instrument calibrations.

#### 4. Datum Systems

The variability of the process pressure datum systems must be held to very close limits, since these datum systems control process pressures.

Much work has been done to control the datum systems. In particular, plantwide datum pressure changes caused by ambient temperature changes have been largely eliminated through the installation of a new type instrument control of the datum pressure.

Since the datum variability remains the largest known contributor to the cascade inventory uncertainty, work is proceeding to determine more closely the datum variability and to determine the effect of datum calibrations on the cascade balance.

### 5. Cascade Streams

Based on present feed schedules, the cascade is fed about 3500 kgms. X in a year, and about the same amount is withdrawn as waste and product. The streams, therefore, total yearly about 7000 kgms. X. It can be easily seen that small biases in stream measurements, weight, purity and assay, have a large contribution to the yearly balance uncertainty; for example, a bias uncertainty of  $\pm$  0.35% in normal feed assay can cause a yearly uncertainty of about  $\pm$  12 kgms. X on that stream alone.

Much work has been done to determine and reduce the uncertainty of all stream measurements. In particular, uncertainties introduced by sampling

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of feed and waste for chemical analysis, the bias in normal feed K-assay, the bias in the ratio of waste to feed K - assay, and the bias in the chemical analysis of the product are the largest stream contributors to the yearly balance uncertainty.

As it may be noted, cascade material balances offer many variables and great masses of data. To interpret results, to correctly weigh the importance of variables, and to take advantage of the great number of measurements, statistics has been employed. In particular, propagation or error and analysis of variance has been used to advantage. In this manner the variables contributing most to the cascade balance uncertainty have been variables contributing most to the cascade balance uncertainty have been determined, and many of the proper steps, such as required number of measurements and required instrument calibrations prior to inventory time, have been taken. It is expected that statistical quality controls applied to many of the measurements will assure consistent, reliable results.

Table I gives present and target estimates of the limit of error and variance of cascade consumption, stream, and inventory variables. To understand the table, a brief explanation of the nomenclature is necessary.

Limit of error is the range in which the true value is believed to be with 95% certainty. Precision refers to the reproducibility of a measurement; bias refers to the systematic difference between the average of a large number of means and the true value. In particular, it is to be noted that a bias is a constant error. No better knowledge of a bias is obtained by repeated measurements. Unless otherwise stated, the given limits of error are in percent of the value of the measurement.

Variance is a square function, thus the unit kg X is used as a weighted linear indication of uncertainty. For example, the current variance of cascade consumption is 2500 kg2X and the current variance of product X-assay bias (laboratory contribution) is 1.00 kg2X; this means that consumption contributes 2500 times as much as the product X-assay bias to the total uncertainty of the annual balance. Variances therefore, are used to judge the importance of the variable uncertainty.

In addition to the work on the cascade balance, progress on the Pure Storage and Laboratory balances has been made. Work on the Recovery, Conversion, Engineering Development and Cont minated Storage balances is in progress.

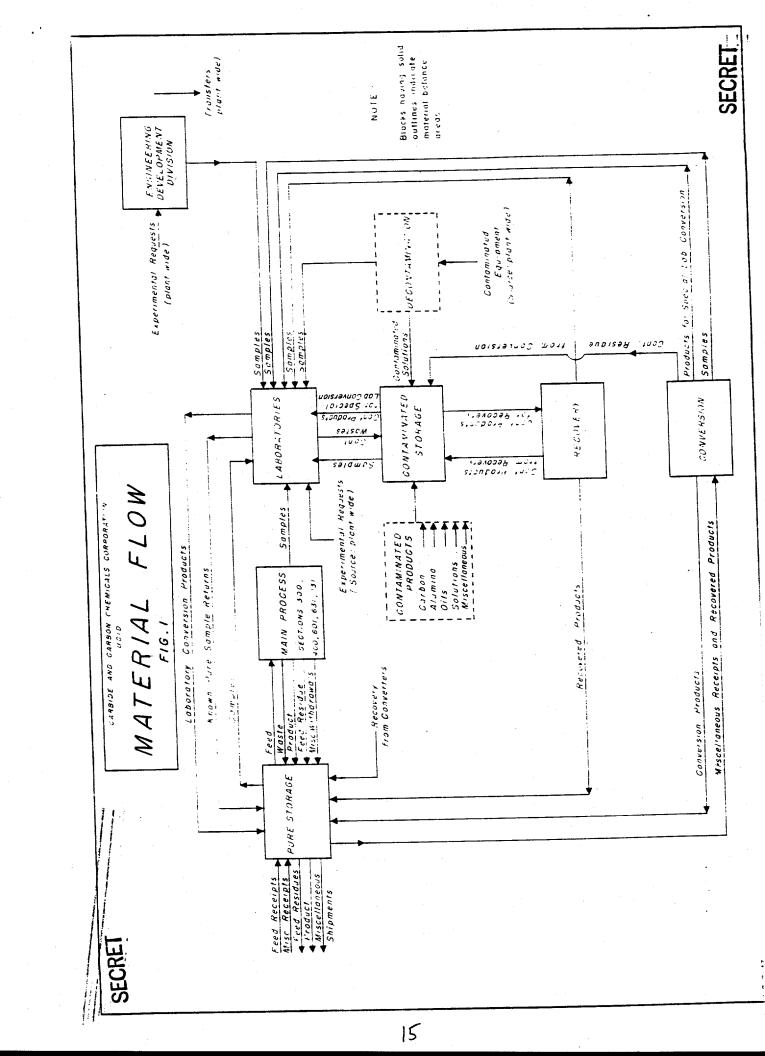


TABLE I

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		Source					0 150	0.02	0.02
Consumption				beed.	Frecision	0.15%	% 60°0	0.65	2.15
Cascade	<u> </u>	T-Analysis	Laboratory Contribution		Bias Frecision		0.15	0.01	10.01
Streams				Waste .	Bias		60.0	0.00	5.5
				Product.	Precision	0.50	0°0	4.50	0.15
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			Contribution	Waste Froduct	Frecision	0.40	0.40	3.40	1.10
		1.000	Laboratory	Peed	Bias Frecition		0.40	20.0	0.06 0.18
		Successive V	Contribution.	Maste/Feed	Bias		60 <b>.</b> 0	0.75	0.60
				Froduct.	Frecision		0.22	1.00	1.00
					Bias	0.00	0.40	0.07	90.0
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Cascade		Factors		Heavy Contaminant	Precision		ς <i>Ē</i> ċ	0.24	0.06
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### PROGRAM AND PROGRESS OF CODED CHEMICALS SECTION - 2. H. ANDERSON

The Coded Chemicals Section is at present rearranging its systems so that an adequate accounting for uranium materials may be made. This appears possible with the solution of four contributing problems, namely proper handling, proper application of laboratory results, proper accounting, and proper auditing of all work.

Procedures standardizing handling have been adopted plantwide. Methods of obtaining and applying laboratory analyses and for accounting are being revised. At this time no procedures have been formulated for auditing. Plant policies for handling and accounting and for the control of samples have been proposed.

Groups to accomplish each of the four unit objectives have been formed. Personnel have been obtained, oriented and trained for three of the unit groups, and personnel are being obtained for the fourth. The following presentation lists accomplishments and current problems of each unit.

### Material Handling Unit

Technical and operations personnel have been set up to control all movements of uranium material outside the cascade. This group requests procurement, receives, warehouses and transfers all such material.

Paramount in importance is the problem of obtaining correct weights and preparing proper transfer papers. The solution of this problem is being developed by obtaining standard non-breakable containers numbered properly and controlling them both when empty and when full. In this manner accurate gross and tare weights are obtained and by difference, the net weight. While close weight agreement is required between sender and receiver, the Coded Chemicals weight is considered the standard, and is used by the accounting section.

Classification of uranium materials in a manner which will permit ready identification aids appreciably in both handling and accounting. A plant wide code system has been proposed by which uranium materials are identified by a number, the prefix of which indicates an X concentration range and the suffix of which denotes the compound type.

### Laboratory Coordination Unit

Technical and clerical personnel have been set up to coordinate requests for analyses, the material to be analyzed and the reporting of this data. This job is difficult because of the detail involved, particularly in adjusting reported results to the present reported feed concentration and in making numerous other conversions to get past results to a unit base.

SECRET

#### Accounting Section

Accountants and clerical personnel have been set up to produce reliable reports on T and X because. This group had previously developed methods of control of stock in numerous containers, and this background was the basis of expansion to control uranium materials plantwide. Ordinary material accounting methods have proven to be inadequate for T and X accounting. This accounting section has been faced with the problem of carrying on existing work by means of the existing material accounting system and at the same time changing system to a double entry fiscal-like system based on T and X. This section keeps three types of information. The first is a balance by vendors on material received, and a balance by material on material received and shipped. This tells how much uranium should be at K-25. The second type is a balance on each area receiving or producing material, kept in detail by ledger accounts. The third is very detailed information on material at each area where material is stored. This is done by a locator system.

#### Audit Section

Personnel are being procured and trained but coordinated work by this section has not been started at this time.

S-E-C-R ET

### HISTORY OF SPECIAL HAZARDS WORK AT K-25

The designing and operating companies of K-25 have been continually conscious of the problem of avoiding a chain reaction in the plant. A brief history is here presented of the groups and personnel concerned with special hazaris and some of the major developments on this subject:

- 1. Dr. E. Teller of Los Alamos served as consultant with The Kellex Corporation and Carbide and Carbon Chemicals Corporation during the design and construction of the plant. At the time the plant was designed, it was contemplated reaching a maximum X concentration of 3680002 in the product. All equipment was therefore designed on this assumption.
- 2. The K-25 Special Hazards Committee was organized in 1944 consisting of the Plant Superintendent and heads of various major departments for consideration of special hazards problems. This committee is still functioning.
  - a. Dr. F.C. Hoyt was appointed as special hazards consultant to the K-25 Plant.
  - b. A working committee was established in the plant to cope with individual operational problems.

The working committee examined operating equipment and procedures, requested necessary investigations, and issued safety rules. Normal and emergency operating procedures were standardized about this time which served to minimize the possibility of hazardous condensations.

- 3. Worthy of special mention because the condensation of TF6 offers a major hazard is the wide elimination of cold trapping operations in the plant which was started in 1945. An alternative method of purging was developed, which eliminated the major items of plant equipment that could be considered dangerous.
- 4. As the X concentration was raised in the product, the need increased for additional information on margins of safety and various factors affecting values of critical accumulations. A series of experiments were conducted in 1946 to determine the values of critical masses under conditions pertinent to the K-25 Plant and the effect of various factors. The experiments were a joint effort of the various companies located in Oak Ridge under the direction of Dr. C. K. Brek of K-25. Experimental values of critical mass for 51480003, 81480008, and 3680008 X were determined under numerous conditions relevant to the K-25 Plant.

- 5. In the summer of 1946, an educational program on critical mass problems was conducted in the plant for both technical and non-technical supervisory personnel who needed the information to perform their work safely. Dr. Back's report (1) on special hazards information was given wide distribution in the plant.
- 6. As a prerequisite to raising the product purity to its present value, a review of K-25 operating equipment and procedures from the special hazards viewpoint was made July 1946 by several groups (2) (3). The following conclusions were reached:
  - a. Operation at high purity could be accomplished safely.
  - b. Several problems required correction before the concentration was increased. (These corrective measures were taken.)
- In order to review and approve all future proposed equipment and procedural changes in Plant II, a body was formed July 29, 1946, known as the Plant II Special Hazards Committee (4).
- 8. Specialinstruments were placed in service.
  - Instruments were installed to detect excessive accumulations of HF which is conducive to attaining a critical situation in case of condensation of both TF and HF.
  - b. Radiation meters were installed throughout the plant to indicate a condition that has become barely critical. Instructions were issued to all operating personnel for evacuating any hazardous area that might develop and for isolating the area with the mid of radiation detectors.
  - 9. Proliminary to raising the product purity in K-25, the Bradbury-Folback-Knith Committee was appointed by the District Office for final recommendations on operating the plant at high purity. A summary of the conclusions of the Committee were:
    - The plant appeared safe for high purity production.
    - b. Safety for future operation depends on a rigorous safety program of continual review of operations by a special hazards group of the operating company, composed of competent personnal, including experienced physicists.

(2) Memo to S. C. Barnett from C. E. Newlon, 8-2-46.

(4) Momo No. 48, Special Hazards Committee For Plant II, S. C. Barnett 7-29-46.

<sup>(1)</sup> Discussion of Special Hazards Safety Fules, C. K. Beck, 6-26-46.

<sup>(3)</sup> Brief Description of Equipment and Procedures in the K-25 Plant Relevant to the Possibilities of Critical Accumulations, C. K. Book, August 15-25, 1946.

- c. Material balances were important in order to follow possible accumulations of X.
- 10. Mr. C. N. Rucker, Assistant Plant Superintendent, outlined on November 1, 1946, the organization to cope with special hazards in the plant. Essentially, it was proposed to continue the operation of the K-25 Special Hazards Committee (Chairman, Dr. C. K. Beck), the Plant Special Hazards Committee (now a working committee with Mr. S. Visner as Chairman); a Radiation Monitoring Group, and to secure the consulting services of several prominant physicists experienced in this field:
- 11. The Special Hazards Section of the Uranium Control and Inspection Department was activated on December 2, 1946. Its function was to devote full time to and coordinate all critical hazards work at K+25 with the exception of certain experimental work.

### PROGRAM AND PROGRESS OF SPECIAL MAZARDS STOTION - S. VISNER

#### Program

The Special Hazards Section has the primary responsibility of coordinating special hazards work in K-25. The following are the objectives of this section:

- 1. To effect the review of proposed changes in plant procedures and equipment from the critical hazards standpoint:
- 21 To effect the review of present procedures and equipment with the purpose of eliminating any possibility of reaching a hazardous condition either through accident or mis-operation.
- 3. To provide adequate radiation monitoring in the plant in cooperation with the Radiation Survey Group of Gascade Services Department.
- 4. To promote educational programs on critical hazards for plant personnel.
- 5. To secure instruments for detection of sul-critical accumulations in the plant.

As a general policy, the "Always Safe" amounts and "Always Safe" diameters serve as the basis for mandling the condensation of process material.

#### Progress

### A. Radiation Monitoring

### 1. Decontamination Tank Radiation

The B-radiation of tolerance levels detected by the Radiation Survey Group of Cascade Services Department about a decontamination tank was attributed to the washing out of quantities of UK from feed and assect inders. Recommendations were made calling for more frequent surveys and dealy scrubbing of the tank tops.

### 2. Product Handling Health Hazards

An investigation is underway to determine possible health hazards to personnel handling plant product due to the high toxicity of U-234 in the product.

# 3. High Pressure Argon Ionization Chambers For Radiation Monitoring

The Instrument Division was requested to construct and test two battery operated High Pressure Argon Ionization Chambers similar to the model procured from Chicago. These two instruments are to be located in the Cold Trap Room and Purge and Product Room in

- 2. The construction of facilities for transporting eight product cylinders in a truck was approved based on spacings calculated by this section.
  - 3. A plan for storing the "Y" cubes in the K-306-7 vault was approved subject to reconsideration whenever the Product Withdrawal Station becomes operative on the cell floor at that location.
  - 4. Operation of a building or cell on direct recycle for purging light contaminants was approved under the condition that the B outlet line from the bottom of the building or cell remain open to the cascade. This is to eliminate the possibility of condensation. Special cases, where it was not feasible to open the B outlet line, would have to be studied individually.

#### D. Miscellaneous

"Safe" spacings were calculated for equipment in the proposed product withdrawal station and for the associated storage cages.

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## HISTORY OF CONSUMPTION AND CORPOSION PROBLETS.

### Laboratory Studies

The choice of The as the process gas for the K-25 caseous Diffusion Process was made for remons pertaining to the physical properties of the gas and not particularly because of its corrosive effects. In fact, information on the resistance of various materials of construction to chemical corrosion by The was totally, or almost totally, lacking. The consumption of process gas represented by this chemical corrosion could materially affect the productivity of any gaseous diffusion plant, therefore, it was recognized that the choice of materials of construction was a vital factor in design.

An extensive laboratory program was launched before the design of the plant was frozen to provide the corrosion information. The consumption rate investigations undertaken by the laboratories (SAM Labs, Bell Telephone Labs, Princeton Labs, Standard Oil of New Jersey Labs, Kellex Labs), have been reported fully by the laboratories themselves. Mr. A. Squires and associates (Kellex Corporation), were forced to extrapolate the meagre information available when the laboratory programs were but partially completed in order to choose materials of construction and determine the effect on expected production rate of X. ("Surface and Consumption of PG, K-25 Plant", pected production rate of X. ("Surface and Consumption of PG, K-25 Plant", A-9-44, Report No. RB-18a, A.N. Squires, et al.")

Early in 1946 at the suggestion of Dr. M. Benedict, a section was formed in the Process Design and Development Department to continue an engineering study of the corrosion and consumption problem in conjunction with the Research Laboratory at K-25. A critical survey, made by this section, of all available consumption data revealed basic weaknesses therein, notably that samples were treated statically and not in dynamic systems comparable to the plant and the specimen pre-treatments with fluorine or exposure conditions varied widely from plant practice. As a result, few data were retained as being applicable for plant consumption estimates. However, the laboratory data did serve to classify materials of construction as to the resistance of each to reaction with  ${
m TF}_6$  and to permit an estimate of the order of magnitude of the plant consumption. The better materials of construction were indicated to be nickel, monel, copper and bronze (in that order), while steels, silver and cadmium were unsuitable. The estimated plant consumption was 9.5 to 18.8 kg. of TF6 per day for K-25 alone. eral reports covering the critical survey and a re-estimate of plant consumption were issued by the Consumption Section. This survey predicted a much lower consumption rate than Squires (1.1-2.8 kg. TF6 per day). mated Barrier Consumption in K-25 and K-27 - Lit. Survey of SAM Reports", 2-20-46, Report No. 1.73.2 (a), C.P. Coughlen; "Estimated Barrier Consumption in K-25 and K-27 - Lit. Survey of BTL Reports", 3-5-46, Report No. 1.73.2 (b), C.P. Coughlen; "Laboratory PG Consumption Rates by Various Metals (Prelim.), 6-21-46, Report No. 1-73-2 (c), C.P. Coughlen; "Review of Surface Consumption of PG in Plant", 7-11-46, Report No. 1.73.8, C.P. Coughlen and D.H. Stewart.")

### Flanning of Corrosion and Consumption Frogram

The hypothesis was adopted that plant consumption is divided into two classes:

1. Static consumption due to exposing materials of construction to nonflowing TF6.

2. Added consumption due to dynamic gas flow and possible reactions with process gas diluents such as moist air.

Since laboratory data, in addition to being insufficient in number, provided information on static consumption only, it became apparent that complete plant consumption information could be obtained only from the plant.

Accordingly, a program to obtain the information was designed. The method of isolating a cell and following consumption by determination of residual TF6 was considered and discarded as unjustifiable for any but 312 Section. cells since the expected magnitude of consumption could scarcely bring about changes in TF concentrations within the ability to determine them. Considerable work has been done in this fashion on 312 Section cells. ("Definition of Problem", 3-12-45; "Statement of Froblem and Outline of Frogram", 2-4-46, Report No. 1.73.1, C. F. Coughlen.) It now appears that similar work in 306 Section cells may be feasible. A proposal for such work has been submitted.

The procedure adopted for the plant survey was the determination of actual T contents of plant surfaces by analyzing removed equipment, as assemblies, or by sectioning the equipment. Specimening was adopted as the method capable of providing information pertaining not only to total plant consumption but to specific materials of construction, to the temperature coefficient, to the effect of location, to the various mechanisms resulting from the dynamic conditions and to provide information relative to deposits of a possible hazardous quantity of T from the critical hazardous standpoint.

A pilot rum was necessary to enable the Research Laboratory to develop the new techniques required by the unprecedented analytical domands and to provide a basis for a proper sampling schedule for the plant scan.

Two consumption rates \* are required; the average rate to any arbitrary date and the current rate. The former is necessary for accounting checks and the latter is necessary for productivity calculations and current accounting checks. The two together provide useful information relative to the ratio of the initial to current consumption rate.

The program was later altered to include the determination by a one-shot treatment of total T contents of converters. The method consists of reconverting the non-volatile T compounds within a convertor to TF6 by a high

\* The more procise term is "Rate of Uranium Build-up" which is identical for a plant-wide consumption rate but is not identical for a specific surface.

temperature fluoring treatment. The generated TF6 is determined quantitatively and check analyses of representative specimens from the converter are made to develop any residual contents. The method; has proved applicable only to converters!

Work was instigated to check the possible reaction of TF with O2 since certain indications suggested that such a reaction might be occurring although there appears to be no proved mechanism for it. Cascade scans to develop O2 disappearance have been run. A controlled experiment to evaluate possible mechanisms is under way on a laboratory scale.

Two accounts have been authorized by the District Engineer for collecting expenses for the consumption program. HL-20 is for all Research Laboratory expenses and HE-26 is for expenses of Engineering Development Division. Uranium Control and Inspection Division and other departments concerned. Dr. R. H. Wiswall is in charge of the consumption program (HL-20) in the Research Laboratories.

### FROGRAM AND FROGRESS OF CONSUMPTION SECTION - C. P. COUGHLEN

### Implementation of Main Program and Results to Date

On April 22, 1946, the convertor, the B pump and the control valve from 306-1.5.6 were made available. The equipment had enjoyed a normal operating history as indicated by existing records. ("Status and Consumption and Corresion Program", 4-23-46, Report No. 1.73.6, C. F. Coughlen; "Selection of #4 Convertor - Meeting Memorandum", 4-15-46, C. F. Coughlen; "Necessary Steps for Determining Flant PG Consumption", 4-2-46, Report No. 1.73.4 (a) - (b), C. F. Coughlen and M. F. Schwenn; "Discussion of Corresion and Consumption - Minutes of Meeting", 4-1-46, Report No. 1.73.3, C. F. Coughlen.) Work proceeded slowly because of the exploratory nature and much destructive specimening was necessary. The results were published in a series of reports. ("Status Report", 8-8-46, Pepert No. 1.73.11, C. F. Coughlen; "Farts Exposed to FG - #4 Converter", 8-22-46, Report No. 1.73.12, Coughlen; "A-C Fump Surfaces Exposed to C-616", 8-23-46, R. A. Schmidt; "Surface T Contents - No. 4 Equipment", 11-25-46, Report No. 1.73.17, C. F. Coughlen and Dr. R. H. Wiswalle)

The actual results indicated the average consumption for the converter examined was 237.0 milligrams of TF6 per day (maximum) while the average consumption for the B pump examined was 57.2 milligrams per day for the exposure time of 259 days. Nickel did not appear to be a better construction material than monel, commercer or bronze while all materials had apparent consumption rates appreciably greater than predicted from laboratory evidence. Some evidence was obtained of an ambient TO<sub>2</sub>F<sub>2</sub> content in the gas streame. Because of a great demand, the single assembly values were extrapolated to the entire plant. However, it is recognized that the values obtained by this extrapolation are not at all reliable.

In August, 1946, it became necessary to obtain rapidly consumption information for Size 3 equipment and the decision was made to apply the fluorine stripping method. The results for three convertors (305-3.3, exposure time 399 days), was 545, 249 milligrams per day for the converters examined. For one A pump from 305-3.3, the average rate was 91 milligrams of TF<sub>6</sub> per day; for one B pump, 102 milligrams of TF<sub>6</sub> per day. The pumps were treated by acid washing the various parts. ("C-616 Removal from Process Converters for Consumption Study", 9-16-46, Report No. 3.98.1, L. C. Olsen; "Average Consumption Rates - No. 3 Tumps", 11-1-46, Report No. 1.73.16, C. F. Coughlen; "Bias On Unit Consumption Values on Converters", 10-12-46, Report No. 1.73.14, "C. F. Coughlen and D. H. Stewart.)

Similar rates for No. 2 converters are being obtained for two different exposure times by both the uranium stripping and the part-by-part wash methods. Size 2 pumps at two different exposure times are being treated by the part-by-part wash methods. No results are yet reportable.

Additional Size 4 equipment, removed from the cascade for purposes other than this investigation, were made available. Both basic methods are being employed to develop rates.

#### Main Program Plans

It is planned to continue part-by-part wash methods until the entire cascade has been scanned to an extent deemed sufficient for adequate material accounting. Such a program will include the development of consumption rates for each size of equipment at two different exposure times. Check rates will likely be developed by application of the fluorine stripper method. ("Rates of PG Consumption - Long Range Program", 9-30-46, Report No. 1.73.15, C. P. Coughlen; "Consumption Program - Converter Removals", (memorandum), 10-22-46, C. P. Coughlen and D. H. Stewart).

#### Miscellaneous Investigations

- A. Several investigations developing the consumption rate by direct measurement of the residual T in an isolated 312 Section cell, have been made and the results reported by R. Maher. A supplementary report is under preparation by J. L. Crites, ("Conference Notes", 7-3-46, C. P. Coughlen; "Procedure for Consumption Tests in 312", 9-17-46, C. J. Crites).
- B. The possibility of react on between dry O2 and TF by some unknown or unproved mechanism was postulated in May, 1946. Several cascade scans have been made. No final reports have been made although results indicate a real less of oxygen within the cascade. Such a loss translates into TO2F2 formation. A laboratory scale experiment yielded only qualitative results which indicate that under certain conditions dry e2 and TF do react. A more complete experiment is being initiated at this time. ("Suggested Mechanism for PG Consumption", 5-3-46; C. P. Coughlen; "Suggested Procedure for Testing Air Inleakage and C2 Consumption in the Cascade", 5-7-46, D. H. Stewart; "Consumption Experiments", 7-16-46, C. P. Coughlen.
- C. Four converters and one pump from the 312 Section have been disassembled for inspection. The development of exact T contents has been held up for more pressing work. However, T compounds are present in excessive amounts in all the equipment examined. TF<sub>6</sub> exposure concentrations from 85% to 10<sup>-4</sup>% are included. ("Proposed Examination of 312 Equipment", 9-20-46, C. P. Coughlen; "Procedure Removal of Whithead Converters", 11-8-46, C. P. Coughlen).
- D. Removed seals were examined for uranium contents and the data treated statistically. The contents were found to be small and of negligible contribution to cascade losses. ("T Loss Resulting From Seal Failures", 3-8-46, C. P. Coughlen; "T Content of Seals Removed From Cascade", 9-10-46, Report No. 1-73-13, C. P. Coughlen).
- E. Isotopic exchanges between the phases of a heterogeneous system temporarily induce apparent consumption rates of excessive magnitudes. ("Molecular Exchange Tests", 1-17-47, G. J. Crites).

- F. Evaluation of an ambient dust burden within the cascade should offer valuable aid in developing explanations for TF<sub>6</sub> consumption mechanisms. A dust filter was designed and installed in the cascade. One completed run indicates there is a significant dust burden but the analyses necessary to fully develop the interpretations have been postponed for more important work. Mr. R. P. Levy conducted an investigation to determine the solids content of liquid waste TF<sub>6</sub> since such a content would have as its source a dust burden in the waste TF<sub>6</sub> gas. Very little solid material was found ("Plant Dust Burden Analysis", 6-27-46, Report No. 1.73.10, C. P. Coughlen; "Evaluation of Dust Burden in Cascade Waste", 9-5-46, Report No. 3.95.1, R. P. Levy and J. D. Mirkus.)
  - The determination of basic static consumption rates for various metals at the various exposure conditions to be found in this or any other plant, has been planned but has not proceeded beyond the discussion stage. This necessary work will be done as soon as possible. ("Development of Engineering Data on Consumption Rates", 6-28-46, Report No. 1.73.9, C. P. Coughlen.)

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